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12 LEVEL II



COMPATIBILITY OF AIRCRAFT OPERATIONAL  
FLUIDS WITH A GRAPHITE/EPOXY COMPOSITE -  
DEVELOPMENT OF AN EXTERIOR COATING SYSTEM  
AND REMOVER.

Final Rept.

K. G. Clark

Aircraft and Crew Systems Technology Directorate  
NAVAL AIR DEVELOPMENT CENTER  
Warminster, Pennsylvania 18974

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## S U M M A R Y

## INTRODUCTION

The objective of this investigation is the identification of aircraft operational and specialty chemicals which are potentially detrimental to the integrity of organic matrix composites. In this report, results of several studies made with the graphite/epoxy Hercules AS/3501-6 are disclosed. Several alternatives to the problem of paint removal are discussed. (1000)

The work reported herein was conducted under AIRTASK No. ZF54-502-001, Work Unit No. ZM501.

## RESULTS

In order to determine the relative compatibility of representative maintenance fluids on the graphite/epoxy under study, off-axis tensile specimens were statically immersed in each of the test fluids for 10 weeks at room temperature, then tested at 250°F (121°C) for residual tensile strength. Results are as follows:

1. MIL-R-81903 (acid-activated remover) caused complete delamination of each specimen.
2. MIL-R-81294 (epoxy paint remover) reduced specimen tensile strength approximately 30 percent.
3. Water and those fluids containing water reduced tensile strengths 13-20 percent.

In addition, static immersion of similar specimens at 212°F (100°C) for nine months in lube oil, hydraulic fluids, preservatives and grease did not reduce specimen strength.

JP-5 turbine fuel at 218°F (103°C) did not swell composite specimens nor reduce tensile, shear, flexure, or flexural modulus values. Equilibrium absorption was found to be approximately 0.30 percent. The effective diffusion coefficient of the fuel through the composite was found to be  $3 \times 10^{-10} \text{ cm}^2/\text{sec}$ .

Further examination of paint remover components revealed that formic acid caused delamination of graphite/epoxy in less than 18 hours at room temperature and phenol in less than 48 hours at 160°F (71°C). The standard epoxy remover caused excessive plasticization of specimens in a cyclic constant amplitude flexure test in less than four hours. Temperature had a profound influence on the activity of MIL-R-81294 in producing delamination of graphite/epoxy.

A "weak link" paint system (TT-L-32/MIL-C-81773) has been developed for graphite/epoxy which can easily be removed by a simple methylene chloride remover also developed for this project. The remover designated (4-70-1) does not produce significant irreversible shear strength losses in the composite when compared with similar exposures to products containing water.

## CONCLUSIONS

Water and maintenance fluids containing water produce significant plasticization of graphite/epoxy, while most solvents, oils, hydraulic fluids, and fuel cause no significant mechanical losses. Paint removal was found to be a significant problem due to the activity of chemical removers. Removal is complicated by the fact that stripping thermoset coatings from graphite/epoxy is more difficult than stripping from aluminum. A "weak link" coating system using a nitrocellulose primer is, thus far, the best strippable composite coating if used with the simple methylene chloride remover designated 4-70-1.

## RECOMMENDATIONS

It is recommended that confirmational testing with tensile, flexure, compression, fatigue and dynamic mechanical specimens of graphite/epoxy and possibly some adhesive, be made. Following these tests, the nitrocellulose/polyurethane coating system should be field tested on graphite/epoxy aircraft substrates.

## FUTURE WORK

In addition to the confirmational testing noted above, a ternary coating system comprised of an amine-cured epoxy, TT-L-32 nitrocellulose, and MIL-C-81773 polyurethane should be evaluated for both graphite/epoxy and aluminum exterior surfaces. Such a system may render masking of graphite/epoxy unnecessary during stripping or repainting operations, since the entire aircraft exterior surface would be stripped with a mild remover.

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## TABLE OF CONTENTS

	<u>Page No.</u>
SUMMARY . . . . .	i
Introduction. . . . .	i
Results . . . . .	i
Conclusions . . . . .	ii
Recommendations . . . . .	ii
Future Work . . . . .	ii
LIST OF TABLES. . . . .	iv
LIST OF FIGURES . . . . .	v
BACKGROUND. . . . .	1
PHASE I. General Screening of Maintenance Chemicals. . . . .	1
PHASE II. Behavior of Graphite/Epoxy in JP-5 . . . . .	8
PHASE III. Graphite/Epoxy and Paint Removers . . . . .	14
A. Remover Component Effects . . . . .	14
B. Dynamic Flexure Testing . . . . .	16
C. Testing for Moisture - Remover Interaction. . . . .	19
D. Effect of Temperature on Static Exposure. . . . .	21
PHASE IV. Paint Removal from Graphite/Epoxy. . . . .	27
A. Chemical Removers: Aluminum vs. Composite. . . . .	27
B. Alternatives. . . . .	29
RECOMMENDATIONS . . . . .	34
FUTURE WORK . . . . .	34
REFERENCES. . . . .	34

## L I S T O F T A B L E S

<u>Table No.</u>		<u>Page No.</u>
I	Maintenance Fluids. . . . .	2
II	Initial Screening of Maintenance Chemicals. . . . .	5
III	Machining Fluid Exposure. . . . .	6
IV	Petroleum Derivatives Exposure. . . . .	6
V	Statistical Significance of Results . . . . .	6
VI	Swelling in JP-5. . . . .	12
VII	JP-5 Static Immersion . . . . .	13
VIII	Paint Remover Compositions. . . . .	14
IX	Remover Component Effects . . . . .	15
X	Dynamic Flexure Results . . . . .	19
XI	Non-phenolic Remover Exposure . . . . .	26
XII	Paint Removal From Aluminum and Graphite/Epoxy. . . . .	28
XIII	"Weak Link" Coating Systems . . . . .	31
XIV	Candidate Coating Systems . . . . .	32
XV	Formulation 4-70-1. . . . .	33
XVI	4-70-1 Remover Exposure . . . . .	33

## LIST OF FIGURES

<u>Figure No.</u>		<u>Page No.</u>
1	Delamination of Graphite/Epoxy by Acid-activated Remover MIL-R-81903 . . . . .	7
2	JP-5 Exposure Vessel. . . . .	9
3	Sorption of JP-5 by 20-ply Graphite/Epoxy Specimens . .	11
4	Dynamic Flexure Test Setup. . . . .	17
5	Dynamic Loading . . . . .	18
6	Dynamic Flexure Test Results. . . . .	20
7	Environments for Testing Moisture/Remover Interaction with Graphite/Epoxy Tensile Specimens . . . . .	22
8	Tensile Strength Test Results for Specimens Exposed to the Cyclic Environments Shown in Figure 7. . . . .	23
9	Delamination of Graphite/Epoxy Immersed in MIL-R-81294 Epoxy Paint Remover at Various Temperatures . . . . .	25

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## BACKGROUND

Current trends in the design of military as well as some civilian aircraft depend upon extensive use of high modulus fiber-reinforced thermoset and thermoplastic polymers. Unfortunately, the overwhelming majority of maintenance and specialty chemicals were designed for servicing aircraft with aluminum skins. While certain organic matrix composites may perform the same functions as metal aircraft components, their intrinsic properties differ widely from metals. For example, with respect to a service environment, aluminum is irreversibly attacked by salt water, most polymers are not; however, most polymers are chemically degraded by sunlight, aluminum is not. In addition, polymers are sensitive to many organic solvents, aluminum is not. In short, there is a vast chemical difference between polymers, in which atoms are bonded covalently, and metals, which are held together by metallic bonding.

In consideration of these differences, a comprehensive list of maintenance fluids was drawn from reference (a) for a general screening of Hercules AS/3501-6 graphite/epoxy by testing for residual mechanical properties. Several fluids which were chemically similar to one chosen as the test fluid were not used. Table I lists those maintenance fluids tested in this study.

### PHASE I. General Screening of Maintenance Chemicals

In order to identify problem fluids rapidly, the first series of evaluations were performed by static immersion of three Hercules AS/3501-6 graphite epoxy test specimens in each of the fluids from Table I and distilled water for 10 weeks at room temperature. Eight-ply (+ 45) sym.tensile specimens, 0.50 in. (1.27 cm) by 6.0 in. (15 cm), were chosen for two reasons. First, such a configuration results in matrix dominant characteristics; since graphite itself is relatively inert, chemical degradation could be expected at the fiber/matrix interface and in the bulk matrix. And second, off-axis specimens produce minimal scatter in tensile strength determinations.

Specimens were chosen at random from a single 24 in. (61 cm) by 48 in. (122 cm) laminate, using a random number table, and numbered consecutively, so that any series of numbered specimens represented a random sample from the original specimen population. Test results from "exposed" sets could then be compared statistically with results from the control specimens.

With the exception of the machining fluid immersions, specimens were removed after 10 weeks, measured, blotted dry and installed on an Instron test machine within one hour of removal. After an eight-minute equilibration period at the 250°F (121°C) test temperature, the specimens were tested for tensile strength at a cross-head speed of 0.05 in./min. (0.127 cm/min.).

Static immersion of the graphite/epoxy specimens in machining fluid concentrates (as received) and water were carried out at 160°F (71°C) for 10 weeks. Because several of the concentrates contained significant amounts of water, the test specimens were removed from the fluids, blotted dry and baked for one week at 275°F (135°C) to remove water prior to tensile testing at 250°F (121°C).

TABLE I. MAINTENANCE FLUIDS

Solvents

P-D-680(I)	Dry Cleaning Solvent
TT-N-95(II)	Naphtha, Aliphatic
TT-M-261	Methyl Ethyl Ketone, Technical
TT-T-291	Thinner, Paint, Volatile Mineral Spirits (petroleum spirits)
TT-I-734	Isopropyl Alcohol
TT-E-751	Ethyl Acetate
MIL-T-19544	Thinner, Acrylic Nitrocellulose Lacquer
MIL-T-19588	Toluene-Methyl Isobutyl Ketone Mixture
MIL-T-81533	1,1,1 - Trichloroethane (Methyl Chloroform Inhibited, Vapor Degreasing

Cleaners

P-P-560	Polish, Plastic
MIL-C-25769	Cleaning Compound, Aircraft Surface, Alkaline Waterbase
MIL-C-43616	Cleaning Compound, Aircraft Surface
B&B 3100	Proprietary Engine Gas Path Cleaner

Fuel

MIL-T-5624	Turbine Fuel, Aviation (Grade JP-5)
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Preservatives

VV-L-800	Lubricating Oil, General Purpose, Preservative (Water- displacing, Low Temperature)
MIL-C-81309(II)	Corrosion Preventive Compound, Water Displacing, Ultra- thin Film

Oil/Hydraulic Fluids/Grease

MIL-H-5606	Hydraulic Fluid, Petroleum Base, Aircraft, Missile and Ordnance
MIL-L-23699	Lubricating Oil, Aircraft Turbine Engines, Synthetic Base
MIL-G-81322	Grease, Aircraft, General Purpose, Wide Temperature Range
MIL-H-83282	Hydraulic Fluid, Fire Resistant, Synthetic Hydrocarbon Base, Aircraft

TABLE I. MAINTENANCE FLUIDS (CONT'D)

Removers

MIL-R-81903	Remover, Acid-Activated, for Amine-Cured Epoxy Coating Systems
MIL-R-81294	Remover, Paint, Epoxy System
TT-R-248	Remover, Paint and Lacquer, Solvent Type
MIL-R-81835	Remover, Organic Coating Hot Tank Type
MIL-R-19853	Carbon Removing Compound Agitated Tank

Machining Fluids (Proprietary)

Hangsterfer's HE-2  
 Hangsterfer's 555  
 Cimfree 234  
 Immunol 1809  
 Johnson's TL-131

Others

MIL-C-81706	Chemical Conversion Materials for Coating Aluminum and Aluminum Alloys
O-A-451	Ammonium Hydroxide, Technical
O-S-576	Sodium Bicarbonate, Technical
MIL-S-13727	Sodium Phosphate, Monobasic, Anhydrous, Technical

The petroleum derivatives (some of which seemed to produce unexplained high strength losses) were reevaluated in a nine month 212°F (100°C) static immersion series. Following the exposure, specimens were blotted dry and tested in tension at 250°F (121°C).

### Results (Phase I)

Average residual tensile strength, coefficient of variation, and strength loss (basis = control) for the composite tensile specimens after exposure to all but the machining fluids are shown in Table II.

Tables III and IV list residual tensile strengths for composite specimens exposed to machining fluids at 160°F (71°C) for 10 weeks and petroleum derivatives at 212°F (100°C) for nine months (respectively).

### Conclusions (Phase I)

The data from Tables II, III, and IV can be analyzed using a one-tailed, modified Student's t-test to compare the control sample with a fluid immersion sample. Table V lists those fluids which exhibited significant strength loss effects on Hercules AS/3501-6 according to the level of significance (5% being significant and 1% being highly significant).

In any statistical experiment, some errors are to be expected. It appears that three fluids (W-L-800, MIL-C-81309, MIL-L-23699) produced Type I errors - that is, a significant difference was indicated by statistical analysis when, in reality, no difference between the control and the test samples existed as shown by the retest of the "petroleum derivatives" at 212°F (100°C) for nine months. Since roughly 1.5 Type I errors would be expected, it could be concluded that the experiment was unlucky.

As expected, water caused a number of significant deviations in tensile strength. Excepting the paint removers, all fluids containing water induced strength losses between 13 and 20 percent. Undoubtedly, sorption of contained water and subsequent plasticization of the epoxy matrix accounted for losses of this magnitude. Remover compounds, however, exhibited a range of effects while the acid-activated remover MIL-R-81903 caused delamination of the graphite/epoxy (see Figure 1), the epoxy remover MIL-R-81294, a primary remover for aircraft coating systems, induced excessive strength losses (approximately 30%). The lacquer remover, on the other hand, caused strength losses that could be attributed to the water contained in the formulation. Further investigation of paint remover effects is reported in Phase III.

With the exception of methyl ethyl ketone (MEK), no solvent tested caused a significant strength deviation. MEK and methyl chloroform produced the highest and second highest deviations presumably by a plasticization mechanism which may very well be reversible as with water. It should be noted, however, that since an eight minute equilibration time at 250°F (121°C) was used, specimen solvent content depended in part on solvent volatility - low

TABLE II. INITIAL SCREENING OF MAINTENANCE CHEMICALS

(Room Temperature; 10 Weeks)

	Average Tensile Strength (psi (MPa))	Coefficient of Variation (COV) (%)	Loss (%)
Control	17 967 (123.88)	4.8	-
Water (Distilled)	14 695 (101.32)	2.4	18.2
<b>Solvents</b>			
P-D-680(I)	16 766 (115.60)	0.8	6.7
TT-N-95(II)	17 935 (123.66)	0.9	0.2
TT-M-261	15 468 (106.65)	1.9	13.9
TT-T-291	17 008 (117.27)	1.6	5.3
TT-I-735	17 194 (118.55)	1.7	4.3
TT-E-751	17 105 (117.93)	4.8	4.8
MIL-T-19544	17 013 (117.30)	1.1	5.3
MIL-T-19588	17 492 (120.60)	1.8	2.6
MIL-T-81533	16 516 (113.87)	5.5	8.1
<b>Cleaners (Water Content)</b>			
P-P-560 (23%)	15 105 (104.15)	4.8	15.9
MIL-C-25769 (85%)	15 079 (103.97)	2.4	16.1
MIL-C-43616 (24%)	15 349 (105.83)	6.1	14.6
B&B 3100 (14%)	14 293 (98.55)	3.5	20.4
<b>Fuel</b>			
MIL-T-5624 (JP5)	16 951 (116.87)	1.2	5.7
<b>Preservatives</b>			
VV-L-800	15 459 (106.59)	7.2	14.0
MIL-C-81309(II)	15 544 (107.17)	1.4	13.5
<b>Oils/Hydraulic Fluids/Grease</b>			
MIL-H-5606	16 118 (111.13)	11.9	10.3
MIL-L-23699	16 354 (112.76)	2.9	9.0
MIL-G-81322	-	-	-
MIL-H-83282	16 711 (115.22)	0.7	7.0
<b>Removers (Water Content)</b>			
MIL-R-81903 (10%)	Delaminated		
MIL-R-81294 (5%)	12 643 (87.17)	0.3	29.6
TT-R-248 (8%)	16 180 (111.56)	2.0	9.9
MIL-R-81835 (10%)	14 811 (102.12)	4.1	17.6
MIL-R-19853 (15%)	14 323 (98.75)	4.6	20.3
<b>Others (Water Content)</b>			
MIL-C-81706 (97%)	14 650 (101.01)	2.2	18.5
A-A-451 (98%)	15 105 (104.15)	2.0	15.9
O-S-576 (65%)	15 286 (105.39)	1.4	14.9
MIL-S-13727 (95%)	15 725 (108.42)	3.1	12.5

TABLE III. MACHINING FLUID EXPOSURE

(160°F (71°C); 10 Weeks)

	Average Tensile Strength (psi (MPa))	Coefficient of Variation (COV) (%)	Loss (%)
Control (Distilled Water)	16 620 (114.59)	1.6	-
Hangsterfer's HE-2	18 195 (125.45)	2.0	no loss
Hangsterfer's 555	17 503 (120.68)	1.6	no loss
Cimfree 234	17 127 (118.09)	4.8	no loss
Immunol 1809	17 264 (119.03)	6.8	no loss
Johnson's TL-131	16 659 (114.86)	5.0	no loss

TABLE IV. PETROLEUM DERIVATIVES EXPOSURE

(212°F (100°C); 9 Months)

	Tensile Strength (psi (MPa))	Coefficient of Variation (COV) (%)	Loss (%)
Control (Air)	17 919 (123.55)	5.2	-
VV-L-800	17 874 (123.24)	2.4	0.3
MIL-C-81309	16 816 (115.94)	2.2	6.2
MIL-H-5606	18 698 (128.92)	3.0	no loss
MIL-L-23699	17 349 (119.62)	4.3	3.2
MIL-G-81322	17 075 (117.73)	1.8	4.7
MIL-H-83282	17 480 (120.52)	1.6	2.4

TABLE V. STATISTICAL SIGNIFICANCE OF RESULTS

<u>Significant</u>	<u>Highly Significant</u>
TT-M-261	B&B 3100
P-P-560	* MIL-R-81903
MIL-C-25769	MIL-R-81294
MIL-C-43616	MIL-C-81706
TT-R-248	
MIL-R-81835	
MIL-R-19853	
O-A-451	
O-S-576	* Delamination occurred
MIL-S-13727	

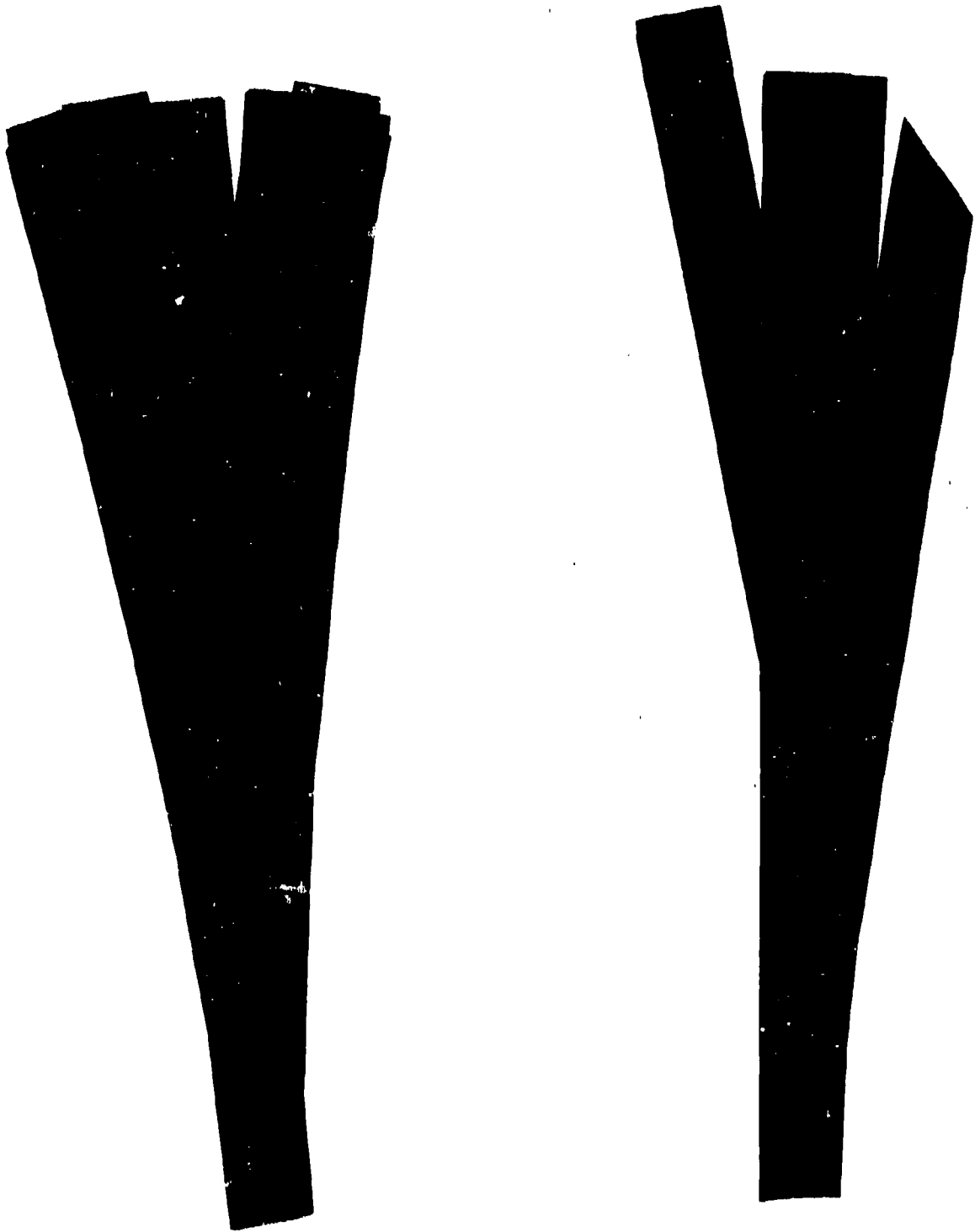


FIGURE 1. DELAMINATION OF GRAPHITE/EPOXY BY ACID-ACTIVATED REMOVER MIL-R-81903

boiling solvents tended to escape the matrix prior to testing. In addition, sorption limits of the various solvents certainly differed as the solubility in the matrix. Overall, however, it appears that organic solvents produce a minor plasticizing effect. Since MEK should contact graphite/epoxy for only a brief drying time interval during painting operations and at much lower concentrations than that used in the static immersion test, such exposures are not considered to be of any consequence.

Long term exposure of graphite/epoxy to "petroleum derivatives" at elevated temperature does not produce any significant strength deviations.

## PHASE II. Behavior of Graphite/Epoxy in JP-5

The design of graphite/epoxy composite wing components for F-18 require jet fuel to be in direct contact with structural laminates at temperatures which conceivably reach 218°F (103°C), the maximum skin temperature for that aircraft. Phase I concluded that organic solvents appear to produce small strength losses at room temperature. The objectives of this study were to confirm the inertness of graphite/epoxy exposed to JP-5 at this temperature with respect to swelling and strength and to measure the rate of diffusion of fuel in a test specimen.

### Experimental (Phase II)

A 1000 milliliter tall-form glass kettle containing JP-5 turbine fuel (MIL-T-5624), which had been dried with elemental sodium to less than 30 parts per million water, ceramic support, test specimens and magnetic stirrer was fitted with a four opening ground glass cover. A thermometer and glass condenser with drying tube were inserted into two cover openings and the remaining two plugged with glass stoppers. The kettle was heated by a magnetic stirrer/hot plate controlled electronically by the thermometer mercury column at  $218^{\circ} \pm 4^{\circ}\text{F}$  ( $103^{\circ} \pm 2^{\circ}\text{C}$ ) (See Figure 2).

Duplicate diffusion specimens were cut from 20-, 10-, and 5-ply unidirectional stock of Hercules AS/3501-6 graphite/epoxy composite. Faces and edges of each specimen were smoothed with number 280 silicon carbide paper to eliminate surface entrapment of significant amounts of diffusant. One specimen of each respective thickness was sealed at each end by 0.5 mil (12.7  $\mu\text{m}$ ) aluminum foil bonded with Hysol EA 9309 structural adhesive to prevent wicking from specimens ends. Swelling measurements were also made with these specimens. Off-axis tensile specimens, 0.5 in. (1.27 cm) by 6.0 in. (15.2 cm) were cut from (+ 45°) 8 ply laminate stock. Unidirectional shear specimens, 0.25 in. (0.64 cm) by 0.6 in. (1.5 cm) were cut from 12-ply stock. Unidirectional flexure specimens, 1.0 in. (2.5 cm) by 2.5 in. (6.4 cm) were cut from 6-ply stock. All test specimens were conditioned in a vacuum oven at 105°C and 10 mm Hg for 96 hours prior to exposure in the fuel.

The effective laminate diffusion coefficient was determined by the method attributed to McKay (reference (b)): if it is assumed that the diffusion coefficient is independent of diffusant concentration, for short exposure times (small  $t$ ),



## JP-5 EXPOSURE

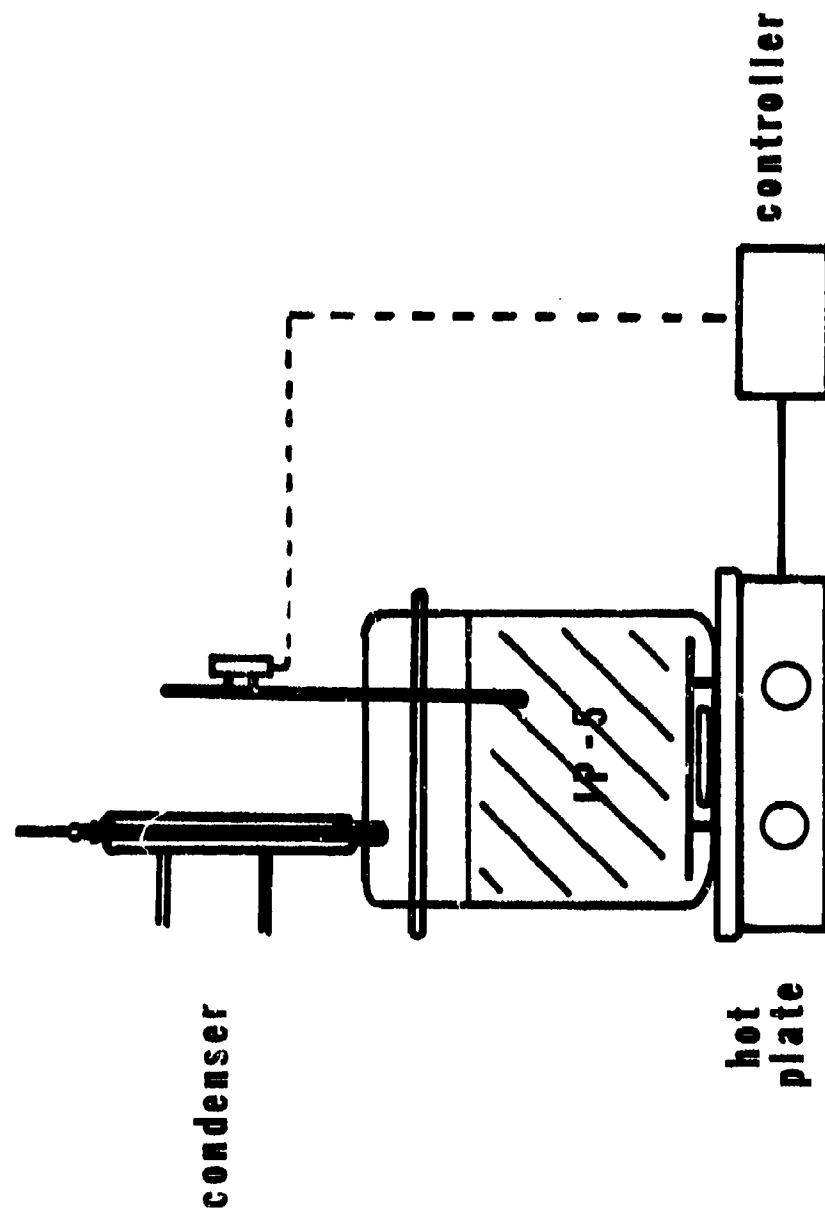


FIGURE 2. JP-5 EXPOSURE VESSEL.

$$(1) \quad D = \frac{\pi l^2 S^2}{16}$$

where S is the initial slope of  $F = (\text{weight of sorbed fluid at time } t) / (\text{weight of sorbed fluid at equilibrium})$  vs.  $t^{1/2}$  and  $l = \text{specimen thickness}$ . Since this plot is reasonably linear to  $F = 0.5$ ,  $D$  can also be determined by locating the half-saturation time  $t(\frac{1}{2})$ ,

$$(2) \quad D \approx 0.04909 \frac{l^2}{t(\frac{1}{2})}$$

The weight and dimension of each diffusion specimen were determined periodically by blotting dry with paper tissue, cooling for five minutes at room temperature, weighing to the nearest 0.1 milligram and measuring specimen dimensions at points designated by scribed marks using a micrometer.

After the 87-day exposure period, the kettle and contents were allowed to cool to room temperature. Mechanical test specimens were blotted dry and tested (within 24 hours of removal from the fuel) at room temperature and 250°F (121°C) (using an 8-minute equilibration time) with the following parameters:

Tensile      Crosshead speed = 0.05 in./min. (0.127 cm/min.)

Short beam shear      Loading radii = 0.125 in. (0.3175 cm)  
                                  Span = 0.4 in. (1.016 cm)  
                                  Crosshead speed = 0.05 in./min. (0.127 cm/min.)

Flexure      Loading radii = 0.125 in. (0.3175 cm)  
                                  Span = 0.625 in. (1.5875 cm)  
                                  Crosshead speed = 0.02 in./min. (0.0508 cm/min.)

### Results (Phase II)

Figure 3 is a plot of the weight gain of the 20-ply diffusion specimens as a function of the square root of time. Early in the experiment, a small sliver was accidentally removed from the sealed specimen causing the line through the data points to be displaced downward. Data obtained with 10- and 5- ply specimens showed greater scatter but appear to reach an equilibrium concentration of approximately 0.30 weight percent in the unsealed specimens. The effective laminate diffusion coefficient as calculated from the equilibrium concentration, the initial slope of the line for the sealed specimen from Figure 2, and equation (1) was determined to be about  $3 \times 10^{-10} \text{ cm}^2/\text{sec}$ . At this rate the 20-ply specimen would have reached half-saturation in 110 days, as determined from equation (2).

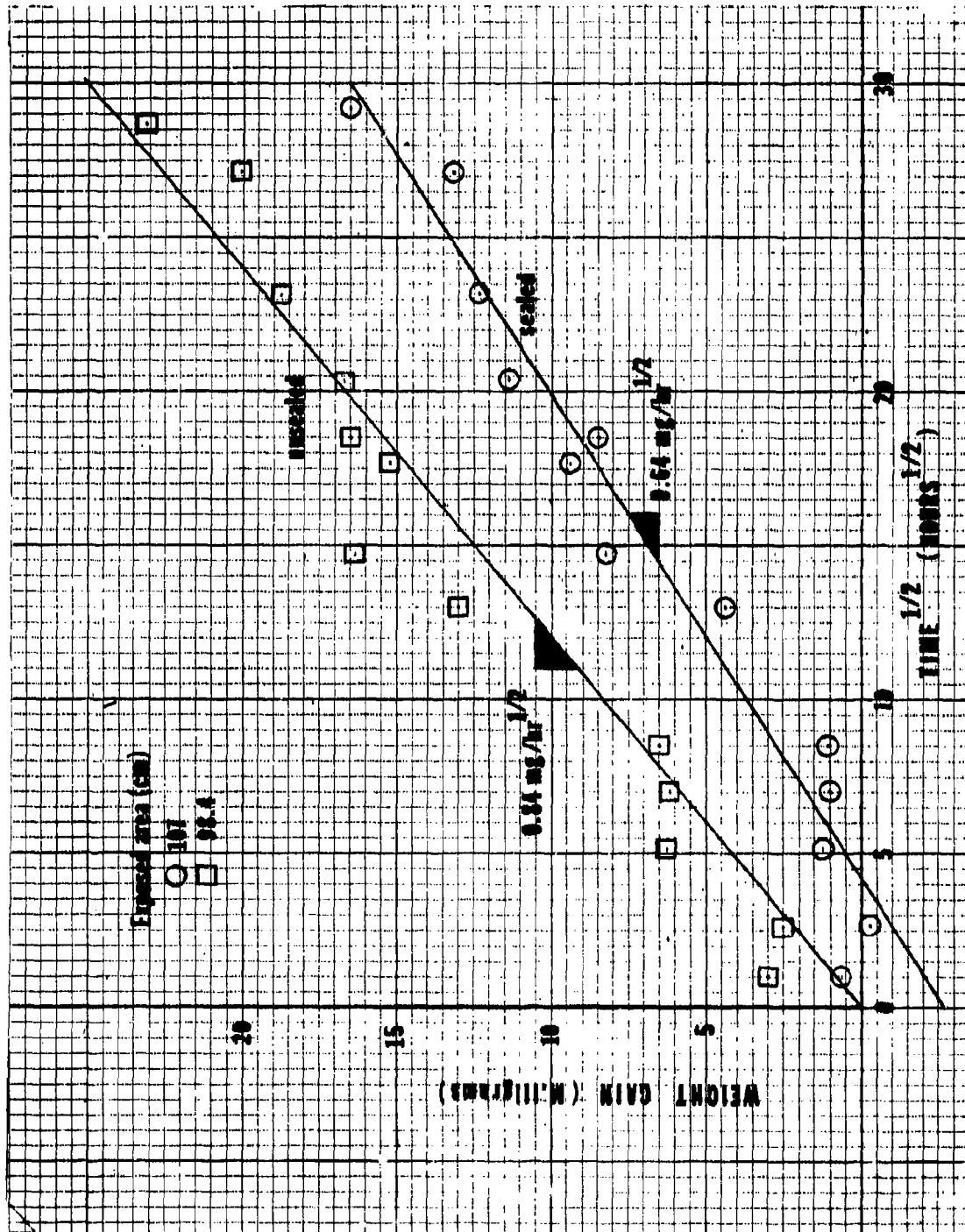


FIGURE 3. SORPTION OF JP-5 BY 20-PLY GRAPHITE/EPOXY SPECIMENS

During the 87-day exposure, a resinous precipitate began to form which became rather pronounced after 900 hours. At that time weight gain measurements were discontinued since significant errors could be expected. Although not detected, this precipitate could have adhered to the surfaces of the diffusion specimens early in the experiment. The value of the diffusion coefficient ( $3 \times 10^{-10}$  cm<sup>2</sup>/sec.), therefore, probably errs on the high side.

Swelling measurements before and after the 87 day, 218°F (103°C) exposure are listed in Table VI.

TABLE VI. SWELLING IN JP-5

	Original (in.(cm))	After Exposure (in.(cm))	(Mils) Change	(%)
20 ply (thickness)	0.0945 (0.2400)	0.0946 (0.2403)	+0.1	(+0.1)
(transverse)	2.668 (6.777)	2.665 (6.769)	-3	(-0.1)
(longitudinal)	-	-		
20 ply (thickness)	0.0948 (0.2408)	0.0948 (0.2408)	0	(0)
(transverse)	2.478 (6.294)	2.478 (6.294)	0	(0)
(longitudinal)	-	-		
10 ply (thickness)	0.0456 (0.1158)	0.0456 (0.1158)	0	(0)
(transverse)	2.679 (6.805)	2.680 (6.807)	+1	(+0.04)
(longitudinal)	2.026 (5.603)	2.025 (5.144)	-1	(-0.05)
10 ply (thickness)	0.0456 (0.1158)	0.0456 (0.1158)	0	(0)
(transverse)	2.029 (5.154)	2.028 (5.151)	-1	(-0.05)
(longitudinal)	2.509 (6.373)	2.507 (6.368)	-2	(-0.08)
5 ply (thickness)	0.0257 (0.0653)	0.0256 (0.0650)	-0.1	(-0.4)
(transverse)	0.913 (2.32)	0.912 (2.32)	-1	(-0.1)
(longitudinal)	2.063 (5.240)	2.065 (5.245)	+2	(+0.1)
5 ply (thickness)	0.0267 (0.0678)	0.0267 (0.0678)	0	(0)
(transverse)	0.997 (2.53)	0.997 (2.53)	0	(0)
(longitudinal)	2.061 (5.23)	2.061 (5.23)	0	(0)
Overall Average	%			
(thickness)	-0.05			
(transverse)	-0.035			
(longitudinal)	-0.0075			

Since the overall average percentages of linear swell indicate decreases in all dimensions, it is evident that dry JP-5 does not induce swelling.

Room temperature and 250°F (121°C) mechanical test results from control specimens (unexposed) and 87-day, 218°F (103°C) JP-5 immersion specimens are listed in Table VII.

TABLE VII. JP-5 STATIC IMMERSION

(218°F (103°C); 87 days)

	<u>Control</u>	<u>*COV</u> <u>(%)</u>	<u>JP-5</u>	<u>*COV</u> <u>(%)</u>	<u>Loss</u> <u>(%)</u>
<u>Room Temperature</u>					
Tensile strength (psi) (MPa)	22 623 (155.98)	3.5	21 459 (147.95)	3.8	5.1
Short beam shear strength (psi) (MPa)	15 365 (103.94)	15	14 717 (101.47)	15	4.2
Flexural strength (psi) (MPa)	237 877 (1640.10)	7.9	224 956 (1531.02)	20	5.4
Flexural modulus (10 <sup>6</sup> psi) (10 <sup>6</sup> MPa)	16.22 (0.1118)	3.4	15.92 (0.1098)	3.4	1.8
<u>250° F</u>					
Tensile strength (psi) (MPa)	17 999 (124.10)	4.8	17 421 (120.11)	4.2	3.2
Short beam shear strength (psi) (MPa)	11 785 (81.25)	4.6	11 386 (78.50)	2.5	3.4
Flexural strength (psi) (MPa)	250 469 (1726.92)	6.1	225 895 (1557.49)	17	9.8
Flexural modulus (10 <sup>6</sup> psi) (10 <sup>6</sup> MPa)	16.07 (0.1108)	3.1	15.31 (0.1056)	3.4	4.7

\*Coefficient of Variation

#### Conclusions (Phase II)

Weight gain measurements for both 10- and 20-ply Hercules AS/3501-6 graphite/epoxy diffusion specimens indicate that end-sealed specimens initially sorb JP-5 fuel at a slower rate than the unsealed specimens. It is quite possible that some wicking occurs from the exposed filament ends. In general, the data for JP-5 diffusion shows much more scatter than that observed in moisture diffusion experiments. The high boiling range of JP-5 (as compared to water) probably delays the evaporation of residual JP-5 from surface entrapment, causing the weight gain to depend in part on the blotting technique.

The effective laminate diffusion coefficient value ( $3 \times 10^{-10}$  cm<sup>2</sup>/sec.) for JP-5 at 218°F (103°C) is approximately 1-2 orders of magnitude less than that for water under the same conditions. It should be noted, however, that the value obtained probably reflects sorption of the lower molecular weight fuel fractions and should not be considered as applying to the entire mixture known as JP-5.

Overall "swelling" percentages actually indicated a shrinkage, but this was below the real detection level estimated at about 0.1 percent. If the composite swells at all in JP-5, it is probably well below this limit.

Finally no significant strength losses were detected in either tensile, short beam shear, flexure or flexural modulus when testing was performed at room temperature and 250°F (121°C). In summary, no deleterious effects were discovered resulting from immersion in JP-5 at 218°F (103°C) for 87 days.

### PHASE III. Graphite/Epoxy and Paint Removers

Paint removers emerge from the Phase I screening as being the single most important problem in maintenance of aircraft fabricated with exterior composite surfaces. In this phase, the importance of the following parameters was studied:

1. Remover components
2. Dynamic loading
3. Moisture
4. Temperature

#### A. Remover Component Effects

Currently, there are five types of paint removers used on exterior aircraft surfaces in the various Naval Air Rework Facilities. Compositions of these removers are listed in order of decreasing activity in Table VIII.

TABLE VIII. PAINT REMOVER COMPOSITIONS  
(Weight percent)

	MIL-R-81903	MIL-R-81294 Phenolic	Non- phenolic	TT-R-248	Rain Erosion Coating Removers
Methylene chloride	64	71	75	44	?
Other solvents	-	-	10	20	?
Phenol	18	20	-	-	?
Cresol	-	-	-	-	?
Formic acid	10	-	-	-	?
Water	2	4	2	7	?
Thickeners, wetting agents, corrosion inhibitors	6	5	13	29	?

Both formic acid and phenol are known as paint remover activators which break chemical bonds to assist in the removal of coatings. Originally, the acid-activated remover MIL-R-81903 was designed to strip amine-cured epoxy coatings which could not be removed with the standard epoxy remover (MIL-R-81294), but due to its high corrosivity (especially on magnesium alloy surfaces) its use has been curtailed. MIL-R-81294 has been the primary paint remover for naval aircraft exteriors, but recent restrictions on phenolic wastes necessitated the inclusion of a slower acting non-phenolic remover in the specification. The TT-R-248 lacquer remover specification forbids the use of phenol and cresol in formulations, but formic acid is not expressly excluded. Although some lacquer removers will eventually strip the Navy paint system from aluminum, there is no specification requirement to this effect. No federal or military specification has been written for rain erosion coating removers; only a few proprietary formulations can strip the Navy paint system from aluminum.

The effect of important remover components was determined using the off-axis tensile specimens as in Phase I. Using 10 weeks or more, room temperature, static immersion exposures, residual tensile strengths were determined at 250°F (121°C). Three specimens, randomly chosen, were used in each fluid, and each was tested within one hour of removal. Results are shown in Table IX.

#### Results (Phase IIIA)

TABLE IX. REMOVER COMPONENT EFFECTS

(Room Temperature, 10 Weeks Minimum)

	Average Tensile Strength (psi (MPa))	* COV (%)	Loss (%)
Control (Air)	17 967 (123.88)	4.8	-
Phenol (90%)	15 683 (108.13)	2.1	12.7
Cresol	17 023 (117.37)	3.0	5.3
Formic Acid (90%)	Delaminated in less than 18 hours.		
** Acetic Acid (99.7%)	16 830 (116.04)	2.0	6.3
Methylene Chloride	17 247 (118.91)	2.7	4.0
** Benzyl Alcohol	17 406 (120.01)	1.1	3.1

\* Coefficient of Variation

\*\* 9-month immersion

Formic acid, a component of the MIL-R-81903 acid stripper, rapidly delaminated the specimens and caused the matrix to take on a deep blue coloration.

After the 10 week phenol immersion, tensile specimens had darkened somewhat, and although visual integrity was good, a significant 12.7 percent loss in tensile strength was found. Reference (c) reports the results of exposure of neat resin samples of Hercules 3501-6 to pure phenol. While exposure at

120°F (49°C) for 360 hours produced a two percent weight loss due to leaching, exposure at 198°F (92°C) resulted in "degradation of the specimen into dark red fragments" within 13 hours. Oddly enough, a composite specimen "maintained integrity after 100 hours of exposure to pure phenol at 92°C."

No other remover components tested produced visual changes in specimens or significant tensile losses.

### Conclusions (Phase IIIA)

Formic acid, which has been used as a reagent in a spot test for bisphenol-A type epoxies, is a powerful nucleophilic reactant which breaks chemical bonds in the epoxy matrix. Obviously, the acid-activated remover MIL-R-81903 should never be used on graphite/epoxy surfaces. It is interesting to note that acetic acid (which differs from formic by a methyl group) causes no significant strength loss after nine months. It has been shown that the epoxy matrix is susceptible to degradation by phenol, especially at elevated temperatures, and that, while no obvious loss of integrity can be observed in composite specimens in room temperature exposures, significant strength losses do occur.

### B. Dynamic Flexure Testing

To determine how rapidly a remover might cause plasticization under conditions of dynamic stress-assisted diffusion, a flexure fatigue-type test was designed. As shown in Figure 4, a six-ply graphite/epoxy flexure specimen, 1.0 in. (2.54 cm) by 2.5 in. (6.35 cm) was deflected at 50 cycles per minute on an Instron test machine. An aluminum dish was provided for immersion of the flexure specimen during the test. Operating parameters were as follows:

Loading radii = 0.125 in. (0.3175 cm)  
 Span = 0.625 in. (1.5875 cm)  
 Crosshead speed = 2.0 in./min. (5.08 cm/min.)  
 Temperature = 74°F (23°C)

The dynamic loading conditions are depicted in Figure 5. Since the limits on flexural strain were fixed by setting maximum and minimum deflection, the corresponding maximum and minimum stress responses as determined from the chart recorder should remain fixed as long as the specimen modulus remains constant. After 90 cycles in air were completed at 2 in./min. (5.08 cm/min.), the crosshead speed was reduced to 0.2 in./min. (0.508 cm/min.) and the recorder turned on for 10 cycles to establish a baseline. Recording at the higher speed was not possible due to the response time of the recorder. The fluid to be tested was then added to the dish, and the specimen was cycled at 2.0 in./min. (5.08 cm/min.) for 90 cycles, then read at 0.2 in./min. (0.508 cm/min.) for 10 cycles. Readings of maximum load were taken in this manner as necessary.



# DYNAMIC IMMERSION

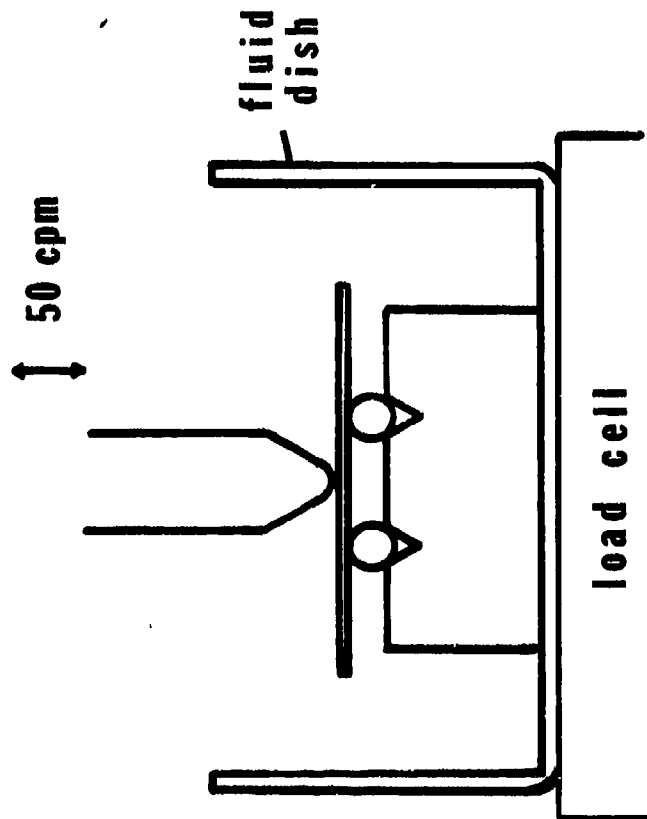


FIGURE 4. DYNAMIC FLEXURE TEST SETUP

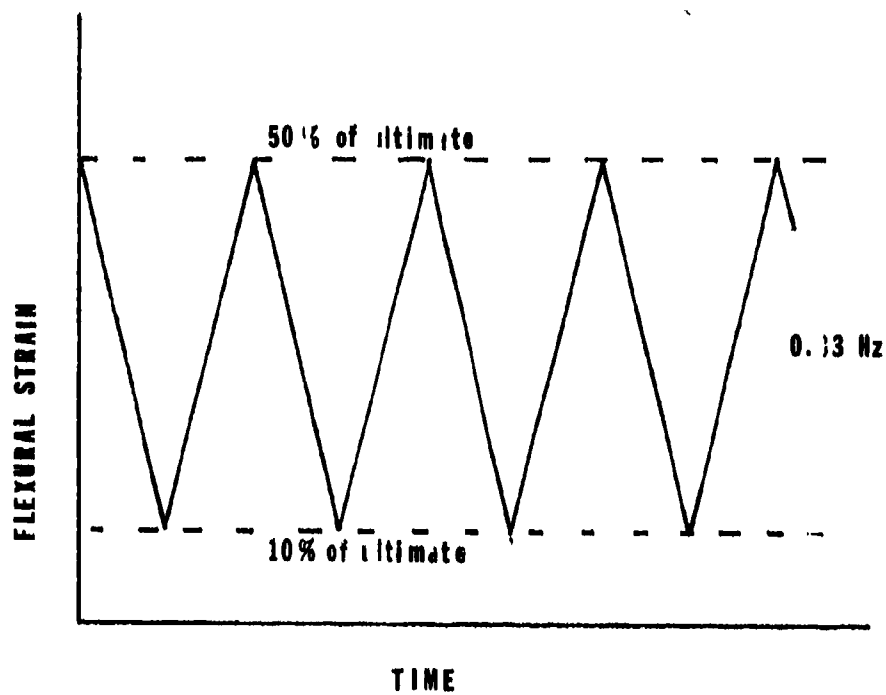


FIGURE 5. DYNAMIC LOADING

Results (Phase IIIB)

Dynamic flexure test results during immersion in the following fluids are reported as decrease in maximum stress response (as a percentage of the initial maximum stress after 90 cycles) versus the number of cycles deflected as plotted in Figure 6.

Air  
Water  
TT-R-248  
MIL-R-81294  
MIL-R-81903

Results are shown in Table X.

TABLE X. DYNAMIC FLEXURE RESULTS

	Percent Decrease in Maximum Stress Response	No. of Cycles	Time (Hrs)
Control (Air)	4	2000	0.7
Water	7	5000	1.7
TT-R-248	18	12000	4
MIL-R-81294	38	16000	5.3
MIL-R-81903	43	12000	4

Conclusions (Phase IIIB)

Although the above results were obtained with no replication, they are considered significant since at least 15 data points were collected for each specimen. Rapid plasticization and/or degradation of thin graphite/epoxy laminates, on the order of time involved in paint removal operations, can occur.

C. Testing for Moisture/Remover Interaction

It is well known that moisture reduces the glass transition temperature of most epoxy resins; Hercules 3501-6 resin is no exception. Presumably, the water molecule hydrogen bonds at polar sites in the epoxy matrix and interferes with intermolecular forces which prevent movement of small segments of the polymer. It is possible that other secondary forces can be disrupted by non-polar components which are present in paint removers. If such is the case, a greater plasticization or strength loss effect resulting from the two types of plasticizer should be evident.

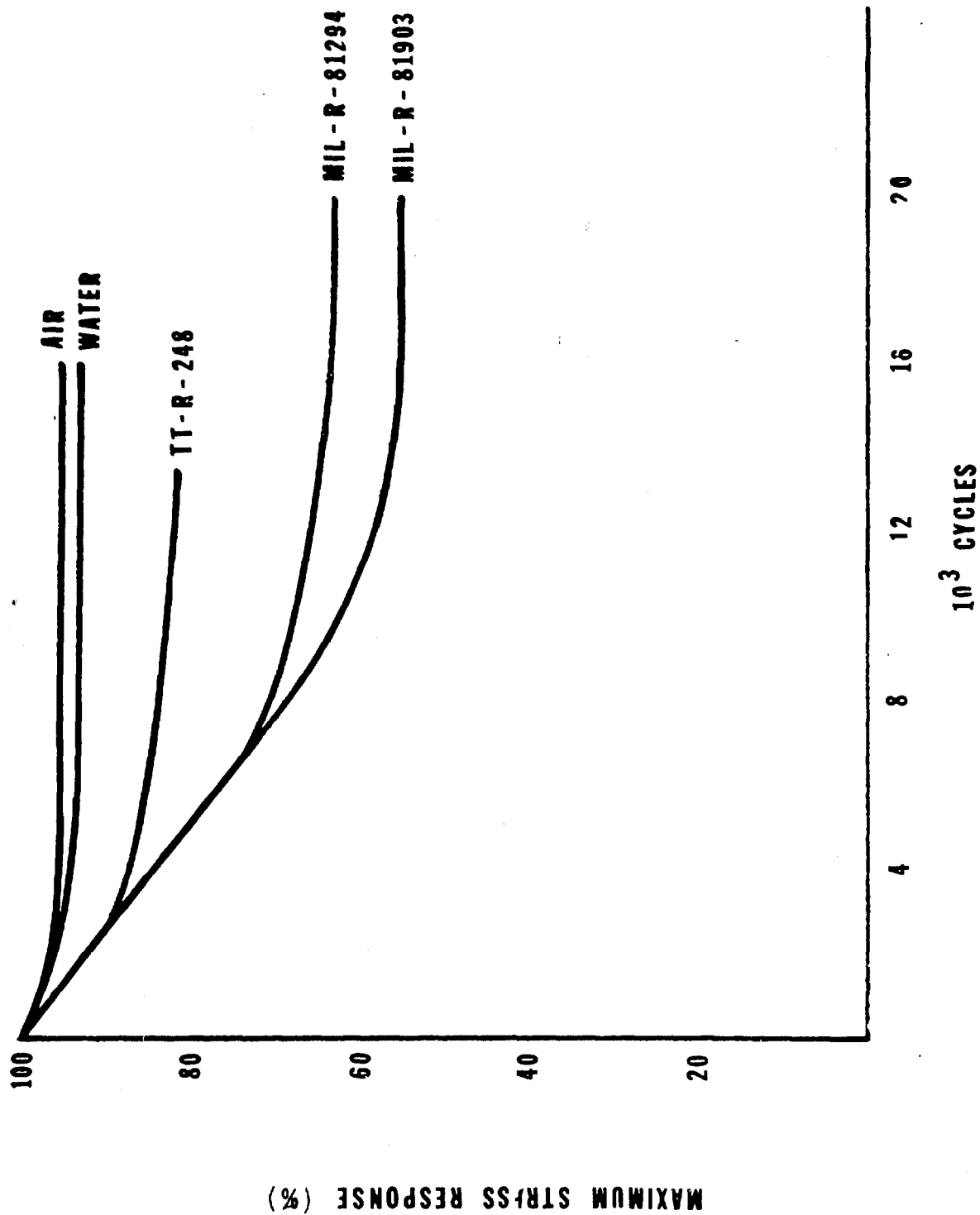


FIGURE 6. DYNAMIC FLEXURE TEST RESULTS

Eight ply ( $\pm 45^\circ$ )<sub>sym</sub> tensile specimens, 0.5 in. (1.27 cm) by 6.0 in. (15.2 cm) were exposed to four cyclic environments as shown in Figure 7. Environment A consisted only of oven drying periods at 212°F (100°C) followed by room temperature storage in a desiccator, B of oven drying followed by immersion in remover at room temperature, C of immersion in boiling water followed by storage at room temperature and 100 percent relative humidity and and D of immersion in boiling water followed by immersion in remover at room temperature. At the end of the first, fourth, and eighth weeks, three specimens from each environment were tested for tensile strength at 250°F (121°C).

### Results (Phase IIIC)

Tensile strength losses are shown in Figure 8. As expected, the dry cycle (A) resulted in consistently high strength. The dry/remover cycle (B) reduced tensile strength approximately 5 percent after the first cycle. After four cycles, still only a 5 percent loss was found. The last two cycles as shown in Figure 7 were actually two weeks long and produced the greatest effect, a loss of approximately 18 percent. The water boil cycle (C) caused a loss of about 18 percent after the first cycle and reached a limiting strength loss of about 25 percent after four cycles. Results obtained with the boil/remover cycle (D) were not significantly different from those with environment (C).

### Conclusions (Phase IIIC)

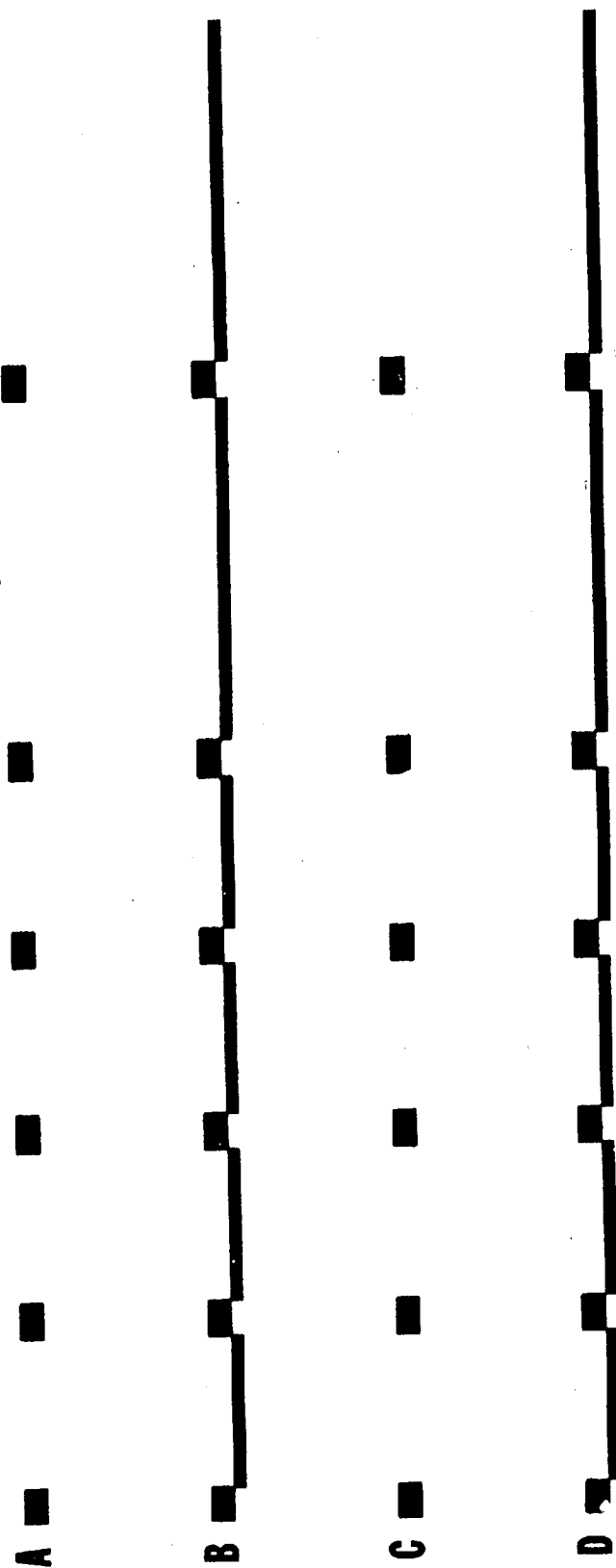
In this experiment, the effect of moisture is apparently greater than that due to the remover, but this is a consequence of the difference in immersion temperatures. When the longer cycles are used, the strength loss due to the remover becomes significant, but more importantly, no corresponding significant change in strength loss was observed for the boil/remover environment (D). It appears that there is a maximum loss due to plasticization of about 25 percent for this graphite/epoxy configuration.

#### D. Effect of Temperature on Static Exposures

The two most important factors in dealing with any given rate-controlled process are time and temperature. Diffusion as well as kinetically-controlled chemical reactions depend heavily on time and temperature. In an effort to determine the worst possible consequence of paint removal operations on Hercules AS/3501-6 graphite/epoxy laminates, tensile specimens, 0.5 inches by 6 inches, were exposed to a typical MIL-R-81294 epoxy remover (see Table VIII) containing phenol at various temperatures - 120, 140, 160, 180, and 200°F (49, 60, 71, 82, and 93°C). Since the major component, methylene chloride, boils at 104°F (40°C), 12-ounce pressure bottles capped with cork-lined tops had to be used. Each bottle was filled to a depth of 2-3 in. (5-7 cm) with the remover, prior to insertion of the test specimens, and stood upright in a vented oven. The bottles were examined periodically for delamination of the specimens.

1 WEEK

1 DAY



DRY AT 212°F **A, B**  
IMMERSION IN MIL-R-81294 AT 75°F **B, D**  
BOIL IN WATER **C, D**

FIGURE 7. ENVIRONMENTS FOR TESTING MOISTURE/REMOVE INTERACTION WITH GRAPHITE/EPOXY TENSILE SPECIMENS

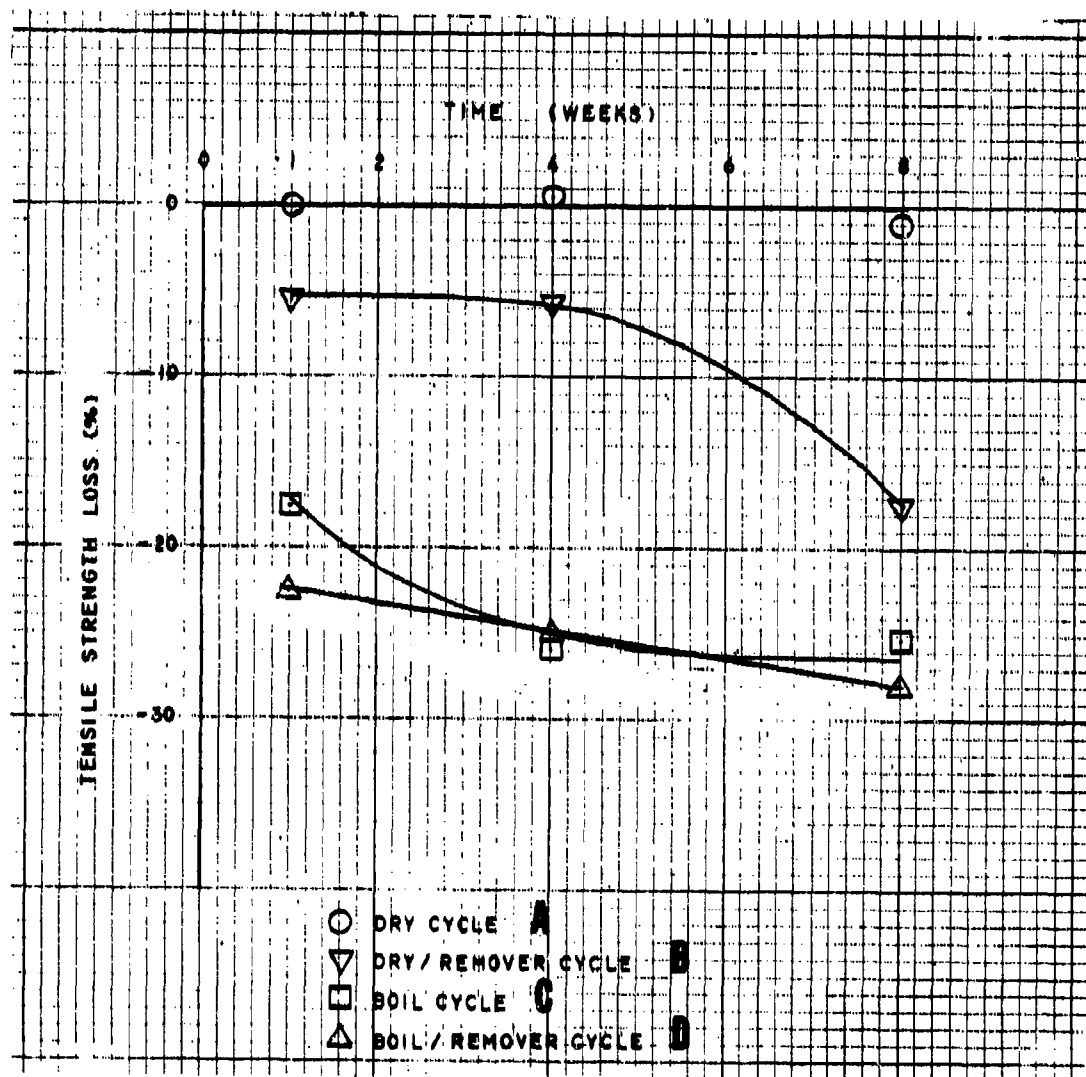


FIGURE 8. TENSILE STRENGTH RESULTS FOR SPECIMENS EXPOSED TO THE CYCLIC ENVIRONMENTS OF FIGURE 7.

Subsequently, similar specimens were exposed to the following remover components in pressure bottles at 160°F (71°C):

Methylene chloride  
 Phenol (90%)  
 Phenol (90%)/Methylene chloride mixture (1 part to 3.5 parts  
                     by weight)  
 Benzyl alcohol  
 Benzyl alcohol/Methylene chloride mixture (1 part to 3.5 parts  
                     by weight)  
 Methanol/Methylene chloride mixture (1 part to 1 part  
                     by weight)  
 Thiophenol/Methylene chloride mixture (2% by weight)

Again, bottles were examined periodically for specimen delamination.

A third series of exposures at 160°F (71°C) was run with the three non-phenolic removers currently listed on the MIL-R-81294 qualified products list. Three tensile specimens and three short beam shear specimens were used. Following a week long exposure, specimens were baked at 275°F (135°C) for one week and tested for tensile and shear strength at 250°F (121°C).

#### Results (Phase IIID)

A crude time-temperature failure region was established as shown in Figure 9. Complete delamination occurred in less than one day at 200, 180, and 160°F (93, 82, and 71°C) but, only started at 140°F (60°C) in about four days and at 120°F (49°C) in about eight days.

Exposure of composite specimens to remover components and mixtures of removers yielded the following results: phenol (90%) and the mixture phenol (90%)/methylene chloride produced one or two ply delamination in less than 48 hours; no other solvent or mixture tested caused delamination or even blistering; however, the methanol/methylene chloride solution did take on a light bluish tint.

Residual tensile and shear strengths of composite specimens exposed to MIL-R-81294 non-phenolic removers are shown in Table XI.

#### Conclusions (Phase IIID)

Previous long term exposure (10 weeks) of graphite/epoxy specimens to MIL-R-81294 epoxy paint remover proved to be mechanically deleterious although no visible deterioration was observed. In this experiment visual manifestations of composite weakening were apparent at 120 to 200°F (49 to 93°C). It is quite possible that pressure buildup (approximately 45 psig (0.31 MPa)) in the bottle accelerated the diffusion of remover components into the specimens, although subsequent chemical reaction of remover and epoxy would not be expected to depend on pressure. As observed in Section A (Remover component effects), phenol is quite harmful to epoxies - the extent to which depends on temperature and time. It has been observed, for example, that at room temperature in MIL-R-81294, Hercules AS/3501-6 will show some delamination of the outer plies after one year.



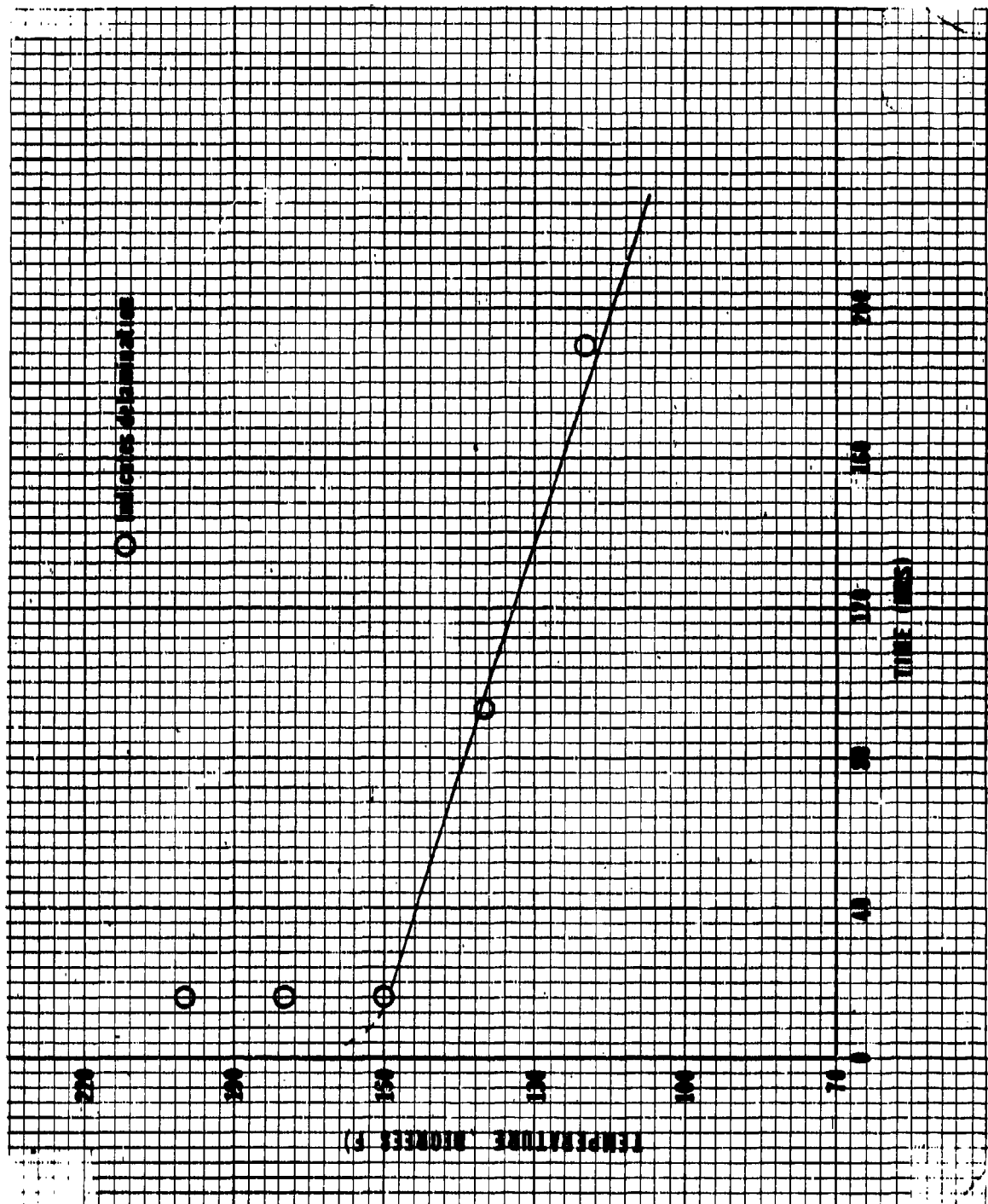


FIGURE 9. DELAMINATION OF GRAPHITE/EPOXY IMMERSSED IN MIL-R-81294 EPOXY PAINT REMOVER AT VARIOUS TEMPERATURES

TABLE XI. NON-PHENOLIC REMOVER EXPOSURE

(1 Week at 160°F (71°C); dried one week at 275°F (135°C))

<u>Tensile Strength</u>	<u>Average Strength (psi (MPa))</u>	<u>COV (%)</u>	<u>Loss (%)</u>
Control (Air)	17 967 (123.88)	4.8	-
Turco 5981 *	11 637 (80.23)	2.8	35.2
B&B 1617A	11 547 (79.61)	2.4	35.7
Eldorado PR 3444	12 565 (86.63)	2.0	30.1

\* Some delamination of top and bottom plies.

<u>Shear Strength</u>			
Control (Air)	11 785 (81.25)	4.6	-
Turco 5981	5 301 (36.55)	3.2	55.0
B&B 1617A	8 666 (59.75)	4.8	26.5
Eldorado PR 3444	9 601 (66.20)	6.0	19.5

Non-phenolic removers can produce outer ply delamination. Mechanical testing of specimens exposed at 160 F (71°C) and dried at 275 F (135°C) to remove plasticizing solvents shows that such exposures are not reversible with respect to strength. Based on confidential formulation data, it would appear that either methanol, sodium chromate, or wetting agents could be responsible for this permanent loss. Additional component testing using mechanical tests after 160 F (71°C) exposure are being planned.

#### PHASE IV. Paint Removal from Graphite/Epoxy

Removal of paint from a graphite/epoxy surface has been studied with the precondition that a standard Navy paint system (MIL-P-23377 epoxy/polyamide primer topcoated with a MIL-C-81773 polyurethane finish) has been applied to a graphite/epoxy surface. In this phase, additional alternatives will be examined under the condition that MIL-C-81773 polyurethane must be the finish coat but with no restriction of primer.

##### A. Chemical Removers - Aluminum vs. Composite

Removability of the standard paint system MIL-P-23377/MIL-C-81773 was evaluated using MIL-R-81294, TT-R-248, and the milder rain erosion coating removers as well as several experimental formulations. Two substrates were used - chemical conversion coated 7075-T6 clad aluminum and Hercules AS/3501-6 graphite/epoxy from which a peel ply sheet had been pulled. The standard paint system was applied and air-dried two months prior to application of paint removers. Stripping times were measured by applying a one-inch diameter spot of a remover on a horizontal painted surface. Wrinkle initiation, half-wrinkle and complete wrinkle times were noted. After 20 minutes, the remover was wiped away with tissue and the percent removal estimated.

##### Results (Phase IVA)

Table XII lists the 20-minute removal percentages for both aluminum and graphite/epoxy surfaces.

The 100 percent figure of Table XII represents complete removal of the topcoat. Primer removal was about 95 percent complete with the residual trapped in surface recesses produced by peel ply removal prior to painting.

##### Conclusion (Phase IVA)

It is apparent that paint removal from composite surfaces is quite different from paint removal from aluminum surfaces. The stripping data from Table XII indicates that phenol may be a requisite component of effective paint strippers for graphite/epoxy, since no removers other than MIL-R-81294 phenolics, Turco 6089, and McGean R256A produced phenolic odors.

TABLE XII. PAINT REMOVAL FROM ALUMINUM AND GRAPHITE/EPOXY

<u>Rain Erosion Coating Removers</u>	<u>Aluminum 7075-T6 (Chemical Conversion Coated)</u>	<u>Graphite/Epoxy (Peel Ply Removed)</u>
Wyandotte 444	100	0
J. B. Moore PR-3	100	0
Mc Gean A-292	100	0
Mc Gean R-256A	100	100 *
Mc Gean A-236	100	0
Turco 6089	100	100 *
Turco 5873	100	0
Turco 6215	100	0
Brulin 810	100	0
TT-R-248	100	0
MIL-R-81294		
Non-phenolic	100	0
Phenolic	100	100 *

\* Distinct phenolic odor; caused delamination of specimens at 160 F (71°C) (tested as in Phase III, Section D).

## B. Alternatives

Three potential solutions to the paint removal problem on graphite/epoxy are:

1. Mechanical paint removal sometimes known as scuff sanding.
2. Use of a diffusion barrier beneath the paint system to prevent remover penetration.
3. Use of a "weak link" coating system, which could be partially removed by a mild remover allowing reapplication of those coatings stripped away.

While mechanical paint removal is simple enough for a small laboratory panel, two major problems could be encountered in actual rework use. First, uneven pressure of sanding belts or disks would probably result in some removal of composite matrix if not fiber. Although the appearance of a greenish yellow color indicates removal of the topcoat, the primer is worn away rapidly due to its relatively low toughness and small thickness. A tougher primer, possibly an amine-cured epoxy coating, sprayed to greater finish thickness could alleviate these problems. The second problem is that, by not completely removing the topcoat, the aircraft coating system could significantly increase in weight by repeated overspraying throughout the life of the aircraft. In addition, the question of priming would have to be considered after an aircraft had been sanded. If the primer thickness had been reduced to a point where its use as an indicator for terminating the sanding operation was doubtful, the aircraft would have to be reprimed. It should be noted that the weight of the MIL-P-23377/MIL-C-81773 paint system is approximately 2.1 pounds per 100 square feet of surface (1.0 kilograms per 10 square meters of surface).

Various metal foils are being studied by airframe manufacturers as diffusion barriers to moisture in the hope of minimizing the problems of moisture pickup. These should likewise prevent the diffusion of harmful remover components into the composite substrates and allow the resulting hybrid to be treated as an exterior metal surface.

A "weak link" coating system depends on the susceptibility of one of the applied coatings to a specific treatment, such as a chemical paint remover, which must be compatible with graphite/epoxy. Early in this program a proprietary rain erosion coating remover (Wyandotte 444) was selected as a potential chemical remover for graphite/epoxy, due to its relatively mild composition. Various experimental coating systems containing a "weak link" coating (susceptible to methylene chloride) were tested for adhesion and strippability. The best system was then further tested for application, physical and chemical properties. Finally, a simple methylene chloride remover was formulated and tested for removal efficiency and effect on short beam shear specimens at 160°F (71°C).

Results (Phase IVB)

"Weak link" coating systems and test results are listed in Table XIII. The elastomeric rain erosion coating exhibited only marginal adhesion when applied over the standard epoxy primer MIL-P-23377. Knife adhesion was only slightly better over the DeSoto Super Koropon primer surfacer. When the rain erosion coating was applied over graphite/epoxy, it could not be removed with the remover (Wyandotte 444). Likewise, polyurethane topcoat MIL-C-81773 could not be removed with the test remover when applied to bare composite substrate. Three systems, an acrylic-nitrocellulose/MIL-C-81773 and two nitrocellulose/MIL-C-81773 systems, showed excellent adhesion and good removability. Results of further tests on these systems are reported in Table XIV. Both the acrylic nitrocellulose and the camouflage nitrocellulose failed the wet tape adhesion test. In addition, it should be noted that the nitrocellulose coatings do not render good adhesion on conversion-coated aluminum.

Table XV lists the formulation for a simple remover capable of stripping the nitrocellulose/polyurethane system.

Short beam shear specimens exposed to methylene chloride, isopropanol (10 weight percent in methylene chloride), formulation 4-70-1, water, and air at 160°F (71°C) in pressure bottles for one week were dried at 275°F (135°C) for one week in a forced draft oven and then tested for shear strength at 250°F (121°C). Results are shown in Table XVI.

Conclusions (Phase IVB)

Coating systems which incorporate the elastomeric rain erosion coating over an epoxy/polyamide primer or an epoxy/amine primer exhibit marginal to fair adhesion which results in good strippability. In these systems, the swelling of the elastomeric coating by the absorption of methylene chloride from the remover and the subsequent generation of high internal stresses in the coating cause it to force itself away from the substrate. Coating systems which incorporate acrylic or acrylic nitrocellulose lacquers do not possess sufficient intercoat or substrate adhesion respectively.

Stripping of acrylic, acrylic-nitrocellulose, and nitrocellulose "primers" occur when methylene chloride swells the polyurethane topcoat and partially dissolves the primer releasing the topcoat film.

A "weak link" coating system using a TT-L-32 (Lacquer, Gloss, for Aircraft Use), nitrocellulose "primer" allows a polyurethane topcoat to be stripped easily from graphite/epoxy substrate using a simple methylene chloride remover. The remover is compatible with graphite/epoxy, based on a statistical comparison of shear strength residuals after exaggerated exposure at 160°F (71°C) in a pressure bottle. For reasons unknown at present, TT-L-20 (Lacquer, Camouflage) nitrocellulose did not pass the wet tape adhesion test and should not be used as a "weak link" primer.

TABLE XIII. "WEAK LINK" COATING SYSTEMS

Substrate	Coating 1	Coating 2	Coating 3	Coating 4	Adhesion (Knife)	Coatings Removed with Wyandotte 444 (Comments)
Aluminum	MIL-P-23377	MIL-C-81773	-	-	Excellent	1-2 (Standard System)
Aluminum	MIL-P-23377	HREC *	MIL-C-81773	-	Marginal	2-3
Aluminum	MIL-P-23377	HREC	MIL-P-23377	MIL-C-81733	Marginal	2-4
GR/E	MIL-P-23377	MIL-C-81773	-	-	Excellent	None (MIL-R-81294 Strips 1-2)
GR/E	Koro **	MIL-P-23377	MIL-C-81773	-	Excellent	None
GR/E	Koro	HREC	MIL-C-81773	-	Fair	2-3 (1 is softened, but dries hard)
GR/E	HREC	MIL-C-81773	-	-	Excellent	None
GR/E	MIL-C-81773	-	-	-	Excellent	None
GR/E	MIL-L-81352	MIL-C-81773	-	-	Poor	1-2
GR/E	MIL-L-19538	MIL-C-81773	-	-	Excellent	1-2
GR/E	TT-L-32	MIL-C-81773	-	-	Excellent	1-2
GR/E	TT-L-20	MIL-C-81773	-	-	Excellent	1-2

\* HREC (Hughson Rain Erosion Coating M413/M200)

\*\* Koro (DeSoto Super Koropon Primer Surfacor)

TABLE XIV. CANDIDATE COATING SYSTEMS

	<u>MIL-L-19538/ MIL-C-81773</u>	<u>TT-L-32/ MIL-C-81773</u>	<u>TT-L-20/ MIL-C-81773</u>
Lifting:			
Topcoated after 15 min.	Pass	Pass	Pass
Topcoated after 30 min.	Pass	Pass	Pass
Topcoated after 1 hour	Pass	Pass	Pass
Scrape Adhesion (kg):			
Primer/substrate	> 10	> 10	> 10
Primer/topcoat	4-1/2	6-1/2	6-1/2
Wet Tape Adhesion:	<u>Fails</u>	Pass	<u>Fails</u>
Fluid Resistance:			
MIL-L-23699 (250°F)	Pass	Pass	Pass
MIL-H-5606 (150°F)	Pass	Pass	Pass
MIL-H-83282 (150°F)	Pass	Pass	Pass
Strippability:			
TT-R-248	1 Min.	2 Min.	2 Min.
NADC Formula 4-70-1	-	1.5 Min.	1.5 Min.
Accelerated Weathering:			
(Xe Arch Weatherometer)	-	Pass	Pass



TABLE XV. FORMULATION 4-70-1

	<u>Grams</u>
Methylene chloride	87.1
Paraffin	1.4
Hydroxypropylmethyl cellulose	1.9
Isopropanol	4.8
Dodacylbenzenesulfonic acid	2.9
Ammonia solution (26%)	<u>1.9</u>
	100.0

TABLE XVI. 4-70-1 REMOVER EXPOSURE

(1 Week at 160°F (71°C); Dried 1 Week at 275°F (135°C))

<u>Shear Strength</u>	<u>Average Strength (psi (MPa))</u>	<u>COV (%)</u>	<u>Loss (%)</u>
Control (Air)	11 466 (79.06)	2.8	-
Water	10 934 (75.39)	4.4	4.6
Methylene chloride	11 809 (81.42)	2.5	-3.0
Isopropanol (10% in MeCl <sub>2</sub> )	11 775 (81.19)	5.4	-2.7
Formulation 4-70-1	10 168 (70.11)	6.6	11.3

## R E C O M M E N D A T I O N S

It is recommended that the TT-L-32/MIL-C-81773 paint system be field tested on graphite/epoxy test sections of an aircraft after the following confirmational tests have been performed:

1. Additional exaggerated exposures of Hercules AS/3501-6 to the remover 4-70-1 should be run with tensile, flexure, compression, fatigue and dynamic mechanical specimens.
2. Similar testing of adhesives which might come into direct contact with the remover or be affected by methylene chloride which has diffused through thin laminates should be performed.

## F U T U R E   W O R K

In addition to the above recommendations, further coatings efforts should address the problem of actual spraying and stripping operations with regard to masking and overspraying. Since TT-L-32 does not adhere well to pretreated aluminum after water exposure, care must be taken to spray this directly on only graphite/epoxy. In addition, since MIL-R-81294 epoxy removers damage composite structures, graphite/epoxy should be masked off during stripping operations. It is possible that such complex masking operations can be avoided by coating the entire aircraft with an amine-cured epoxy to which TT-L-32 should adhere well in wet tape tests. The epoxy base coat could then be coated with TT-L-32 nitrocellulose and MIL-C-81773 polyurethane. All but the epoxy base coat would be easily stripped with 4-70-1 type removers, leaving the amine-cured epoxy intact for subsequent overcoating with fresh nitrocellulose and polyurethane. Such a coating system would eliminate the need to mask any composite surfaces during stripping or any aluminum surfaces during the nitrocellulose application.

## R E F E R E N C E S

- (a) Naval Air Systems Command Technical Manual 01-1A-509, Appendix A of 1 June 1975.
- (b) Mc Kay, A. T., Proc. Phys. Soc. (London) 42, 547 (1930).
- (c) McCullough, R. L., "Influence of External Plasticizers on the Mechanical Properties of Carbon-Fiber Composites," Progress Report N62269-78-66-81106.

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